



ORIGINAL ARTICLE

# Functionalized carbon nanotube doping of P3HT:PCBM photovoltaic devices for enhancing short circuit current and efficiency



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**Abstract** We have successfully functionalized multiwalled carbon nanotubes (MWCNTs) using nitrene approach employing the two aryl azides as a precursor for nitrene generation. The dispersion of functionalized MWCNTs has been enhanced in various organic solvents. These functionalized MWCNTs have been successfully doped in various concentrations in the bulk heterojunction (BHJ) organic photovoltaic (OPV) cells with a poly (3-hexyl thiophene) (P3HT) and [6,6]-phenyl-C<sub>61</sub>-butyric acid methyl ester (PCBM) photoactive blended layer. The incorporation of MWCNTs with aryl functional groups, in active the layer, results in enhanced performance with respect to a reference cell. The maximum power conversion efficiency of 1.86% is achieved with adduct I while in the case of adduct II it gets double to 2.0% in comparison with a reference cell. This improvement in the device performance is attributed to enhanced exciton dissociation and improved charge transport properties due to the formation of a nanotube percolation network in the photoactive composite layer.

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## 1. Introduction

In recent years organic photovoltaic (OPV) devices based on electron-donating conjugated polymers and electron-

accepting fullerene derivatives have emerged as the potential candidate for low-cost and flexible renewable energy sources [1–3]. When light is incident on the devices, it is absorbed by the polymer and excitons are generated on the polymer chains [4,5]. These excitons then diffuse to the polymer-fullerene interface where they dissociate into electron and hole, and the typical diffusion length is about 10 nm [5]. The most successful used OPV material combination is the composite of poly (3-hexyl thiophene) (P3HT) and [6,6]-phenyl-C<sub>61</sub>-butyric acid methyl ester (PCBM), which is now used as the model system and has achieved high efficiencies under solar illumination [6,7].

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Recently, due to their exceptional charge transport properties and electron acceptor behavior carbon nanotubes (CNT) are incorporated into OPVs by doping them into the P3HT/PCBM photoactive layer to utilize them. In photoactive materials, both single walled carbon nanotubes (SWCNTs) and multiwalled carbon nanotubes (MWCNTs) have been incorporated to enhance device performance [8–15]. The introduction of nanotubes in the photoactive layer significantly increases electron mobility thereby reducing the recombination rate and result in enhancing the OPV device performance. One important problem with these devices is the compatibility of different phases in the photoactive layer, predominantly of the nanotubes, which are insoluble in most of the solvents used to fabricate the devices. This low solubility of nanotubes is the main hindrance in obtaining uniform blends of polymer/fullerene/nanotubes which lower the performance of devices. The drawback can be overcome by functionalization of nanotube using suitable reagents, which lead to good dispersibility of functionalized product in organic solvents [16–18].

Due to their low cost of synthesis and easy availability, MWCNTs have found increased application in OPV devices. In the literature, there are reports, using the carboxylic acid functionalized MWCNTs (O-MWCNTs) for improved thin film deposition compared with pristine MWCNTs which leads to enhanced power conversion efficiency of OPV cells [19,20]. Due to the chemical functionalization nanotube bundles exfoliate and this leads to improved solubility and processability. The functionalization modifies the chemical and physical properties of nanotubes thereby improving the better interaction between the nanotubes and polymer [21]. The highest power conversion efficiency obtained by doping functionalized MWCNTs to P3HT/PCBM was 2.5% of under AM1.5 illumination [22].

In this communication, we report functionalization of MWCNTs using two aryl azides, which serve as a precursor for nitrene generation. The incorporation of these functionalized adducts in P3HT/PCBM OPV devices in different doping concentrations, results in improved performance compared to standard devices. These functional groups were chosen such that will tend to form more percolation networks with P3HT and PCBM. Also, due to their enhanced solubility these adducts result in the better blend with both P3HT and PCBM, resulting in shorten exciton dissociation pathway and better device performance.

## 2. Materials and methods

Multi-walled carbon nanotubes (MWCNTs) were grown by the Chemical Vapor Deposition (CVD) method. (Poly (3,4-ethylenedioxythiophene): poly (styrene sulfonate) (PEDOT: PSS), Indium tin oxide (ITO), regioregular poly (3-hexylthiophene) (RR-P3HT), chlorobenzene and thin layer chromatography TLC plates were purchased from Sigma-Aldrich. All the chemicals were used without any purification. Acetone, petroleum ether, 1, 2, dichlorobenzene, dimethylformamide and diethyl ether were purchased locally. All chemicals used were of AR grade.

High-resolution transmission electron microscopy (HR-TEM) was recorded on TECNAI 300 at 300 kV, and Infra red spectra were recorded on Perkin Elmer spectrum 2000 FTIR spectrometer using KBr pellet in range

400–4000  $\text{cm}^{-1}$ . Raman spectra were recorded on Renishaw in via Raman microscope with a polarized laser at 514 nm. Thermal gravimetric analysis (TGA) was recorded on Perkin Elmer Model TGA-7, USA, in nitrogen at a heating rate of 10  $^{\circ}\text{C}/\text{min}$  from 20 to 800  $^{\circ}\text{C}$ . Alumina was used as the reference material with the platinum crucible to carry out the analysis. Fluorescence spectra were recorded on Varian and UV-Vis-NIR spectra were recorded on a UV-1601 Shimadzu instrument in the range 200–1100 nm.

### 2.1. General procedure for the functionalization of nanotubes

It has been reported previously that pre-treating MWCNTs with microwave irradiation leads to a better degree of functionalization; therefore, the MWCNTs were pre-treated with microwave irradiation before covalent modification reaction. In general, 10 mg of the dried material was taken in the reaction vial and subjected to the microwave irradiation in the domestic microwave oven at 100 W for 15 min with 3 min on and one min off cycle.

For covalent functionalization reaction 10 mg of microwave-treated MWCNTs was suspended in a three neck flask with 10 ml of ortho-dichlorobenzene (ODCB) as a solvent and sonicated for the six hours and after that reaction vessel was placed in a pre-heated oil bath at 180  $^{\circ}\text{C}$  (Fig. 1). In another test tube, 110 mg of azide was dissolved in 5 ml of chlorobenzene and added slowly with the help of a syringe to the reaction vessel over a period of one hour. The inert atmosphere was maintained throughout using nitrogen.

The progress of the reaction was monitored using TLC plates for checking decomposition of azide. After four hours no azide could be detected and the reaction was quenched by removing it from heat. The reaction mixture was then allowed to cool to room temperature and subjected to centrifugation to remove carbon nanotubes, which had not reacted or been only partially functionalized at 2000 rpm. The supernatant was collected and product was precipitated using acetone. The precipitated product was collected by centrifuging at 100,000g. The pellet was washed repeatedly with Millipore water, and the final product (3 mg) was dried in the oven at 70  $^{\circ}\text{C}$ .

### 2.2. Fabrication and characterization of photovoltaic cells

The chemically functionalized MWCNTs have been incorporated in the blend of P3HT/PCBM (1:1) in different doping concentrations (1–5%). In the photoactive layer for OPVs, modified MWCNTs of required doping concentration were first dispersed in 1 ml of chlorobenzene and sonicated for 4 h. Then 15 mg of P3HT was added to all solutions, and stirred for 3 h and 15 mg of PCBM was added to the P3HT/MWCNT mixture. These mixtures were stirred overnight and deposited on plasma oxidized ITO/Glass substrates. The spin-coated thin films were allowed to dry for about 40 min at room temperature and then annealed at 125  $^{\circ}\text{C}$  for 10 min. This whole process was carried out in a nitrogen filled glove box. The hole blocking layer bathocuproine (BCP) and the aluminum (Al) electrode were thermally evaporated using a shadow mask under vacuum at the rate of 2  $\text{\AA}/\text{s}$  and 3  $\text{\AA}/\text{s}$ , respectively. Immediately after the device fabrication (Fig. 2), current-voltage measurements were collected using a Keithley 2425 source meter exposing the devices to a 300 W Xe Arc

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